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IS 3389 (1994): Urea-formaldehyde Moulding Materials [PCD 12: Plastics]

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(पहला पुनरीक्षण)

Indian Standard

UREA-FORMALDEHYDE MOULDING MATERIALS — SPECIFICATION

(First Revision)

UDC 678·652' 41' 21·033·3

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1994 Price Group 5

Plastics Sectional Committee, PCD 12

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Urea-formaldehyde (UF) moulding materials are notable for ease of moulding and fabrication. Largely because of unlimited colour possibilities and chemical inertness, they are widely used for the manufacture of a number of popularly used items like cosmetic containers, closures, buttons, radio cabinets, knobs, light weight tableware, toilet seats, electric wiring devices, etc.

This standard was originally published in 1965 Requirements for the flow characteristics of the material was not covered due to non-availability of a suitable test method giving reproducible and repeatable results, although it had been felt to include the requirements at a later date because of its great importance specially on the multi-cavity mould Relaxation in the volume resistivity requirement was also given because of non-availability of proper type/grade of urea required for the plastics industry. Requirements for apparent powder density, density of moulding and bulk factor and mould shrinkage and the limits thereof were left to the agreement between the purchaser and the supplier, permitting the purchaser to use his option for selection to suit his requirements.

This standard was largely based on BS 1322 : 1956 'Specification for aminoplastic moulding materials', issued by the British Standard Institution, which has since been revised after being aligned completely with ISO 2112 : 1977 'Plastics — Aminoplastics moulding materials, issued by the International Organization for Standardization. This revision has been prepared as a result of review of the standard by Thermosetting Moulding Powders and Resins Subcommittee, PCD 12 : 1, in the light of the present day requirements and advancements that have taken place in this field.

While preparing this specification, considerable assistance has been taken from the standards BS 1322:1981/ ISO 2112 : 1977 issued by the British Standards Institution and ISO 2112 : 1990 issued by the International Organization for Standardization.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 JANUARY 2000
TO
IS 3389 : 1994 UREA-FORMALDEHYDE MOULDING MATERIALS — SPECIFICATION

(First Revision)

(*Page* 1, *clause* **2**) — Delete 'IS 2221 : 1962' and insert 'IS 13360 (Part 3/Sec 4) : 1995/ISO 171 : 1980 'Plastics — Methods of testing : Part 3 Physical and dimensional properties, Section 4 Determination of bulk factor of moulding materials' and 'IS 13360 (Part 8/Sec 1) : 1997/ISO 62 : 1980 'Plastics — Methods of testing : Part 8 Permanence/Chemical properties Section 1 Determination of water absorption' after 'IS 13360 (Part 2/Sec 4) : 1993'.

[*Page* 2, *Table* 1, *Sl No.* A (i), *last column*] — Substitute 'IS 13360 (Part 3/Sec 4)/ISO 171 : 1980' *for* '5 of IS 2221 : 1962'.

[*Page* 2, *Table* 1, *Sl No.* B (xi), *last column*] — Substitute 'IS 13360 (Part 8/Sec 1)/ISO 62 : 1980' *for* '8 of IS 2221 : 1962'.

(PCD 12)

Reprography Unit, BIS, New Delhi, India

Indian Standard

UREA-FORMALDEHYDE MOULDING MATERIALS — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for general purpose urea-formaldehyde moulding materials which have been further subdivided into different grades based on property levels and requirements.

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
335 : 1983	New insulating oils (*third revision*)
2221 : 1962	Methods of test for aminoplastic moulding material
2259 : 1963	Methods of test for determination of insulation resistance of solid insulating materials
2584 : 1963	Method of test for electric strength of solid insulating materials at power frequencies
2824 : 1975	Methods for determining the comparative tracking index of solid insulating materials under moist conditions (*first revision*)
2828 : 1964	Glossary of terms used in the plastics industry
13360 (Part 1) : 1992	Plastics — Methods of testing : Part 1 Introduction
13360 (Part 2/Sec 4) : 1993	Plastics — Methods of testing: Part 2 Sampling and preparation of test specimens, Section 4 Preparation of test specimens by machining
13411 : 1992	Glass reinforced polyester dough moulding compounds (DMC)

3 TERMINOLOGY

For the purpose of this standard, the definitions given under **2** of IS 2221 : 1962 and IS 2828 : 1964 shall apply.

4 GRADES

4.1 There shall be three grades as follows:

a) Grade UF A 10	Substantially alpha-cellulose-filled materials superior in electrical and mechanical properties
b) Grade UF A 11	Substantially alpha-cellulose-filled materials, general purpose
c) Grade UF A 20	Substantially wood-flour filled materials

5 REQUIREMENTS

5.1 Description

The material shall be homogeneous consisting of uncured or partially cured urea-formaldehyde resin combined with fillers (cellulosic and wood-flour) and pigments or dyes as required.

5.2 The materials complying with this specification shall meet the appropriate property requirements shown in Table 1.

5.3 It must not be inferred from the above that materials of any particular type are necessarily unsuitable for applications other than those indicated, or that specific materials will be suitable for all applications within the wide description given.

6 TEST SPECIMENS

Bulk factor, flow and volatile matter shall be determined on the moulding material. The other properties shall be determined on moulded test specimens prepared in accordance with Annex A. It is permissible to machine test specimens [IS 13360 (Part 2/Sec 4) : 1993] from sheet moulded in accordance with the moulding conditions of Annex A, as long as it can be shown that the specimens give result which do not differ significantly from those obtained using moulded specimens.

Test specimens to be used for determining the properties given in Table 1 shall be conditioned

Table 1 Requirements for Urea-Formaldehyde Moulding Materials

(*Clause* 5.2)

Sl No.	Property	Units	Grade			Method of Test
			UF A 10	UF A 11	UF A 20	
A.	*Properties measured on moulding powder*					
i)	Bulk factor, *Max*	—	Values to be agreed between the interested parties			5 of IS 2221 : 1962
ii)	Flow	—	do			To be agreed between the interested parties
iii)	Volatile matter, *Max*	%	Value to be agreed between the interested parties	—	—	Annex B
B.	*Properties measured on Test Specimens*					
i)	Flexural stress at rupture, *Min*	MPa	70	60	60	Annex F of IS 13411 : 1992
ii)	Izod impact (Notched) strength, *Min*	KJ/m^2	1·5	1·3	1·3	Annex E of IS 13411 : 1992
iii)	Temperature, deflection under load, *Min*	°C	110	110	95	Annex H of IS 13411 : 1992 (Method A)
iv)	Incandescence resistance	—	Values to be agreed between the interested parties			Annex C
v)	Insulation resistance after 24 h in water, *Min*	ohm	1×10^{10}	1×10^{9}	1×10^{8}	IS 2259 : 1963
iv)	Electric strength, *Min*	MV/m	4·0	3·0	2·5	IS 2584 : 1963
vii)	Comparative tracking resistance (CTI), *Min*	V	CTI 500	CTI 500	CTI 500	IS 2824 : 1975
viii)	Water absorption (Cold), *Max*	mg	175	200	250	Annex D of IS 13411 : 1992 (Method 1)
ix)	Mould shrinkage, *Max*	%	1·0	1·2	1·2	Annex B of IS 13411 : 1992
x)	Post shrinkage, 48 h, *Max*	%	Values to be agreed between the interested parties			do
xi)	Boiling water absorption, *Max*	mg	650	700	750	8 of IS 2221 : 1962

NOTE — The values specified in this table represent the mean result for the property measured.

under prevailing atmospheric conditions as allowed in IS 13360 (Part 1) : 1992 unless otherwise stated in the method of test or agreed to between the interested parties.

Test shall commence not less than 16 h and not more than 72 h after the test specimens have been moulded, unless otherwise specified in the test methods.

When the test specimens have been moulded from powder that has been preheated or dried, then this fact shall be stated in the test report. The conditions of preheating or drying shall also be given.

7 METHODS OF TEST

For mechanical tests, it is permissible for the force to be applied in an alternative direction to that specified in the test methods, provided that the results do not differ significantly from those obtained when the force is applied in the specified direction.

7.1 Determination of Flexural Stress at Rupture

Five test specimens of length not less than 80 mm, of width 10 mm and of thickness 4 mm shall be used.

For both moulded bars and test specimens machined from material moulded in the form of a plate, the load shall be applied parallel to the direction of moulding pressure. The test speed shall be 2 mm/min ± 0·2 mm/min.

Test shall be carried out in accordance with Annex F of IS 13411 : 1992.

7.2 Determination of Izod Impact Strength

Five test specimens shall be used.

In the case of specimens machined from material moulded in the form of a plate, the load shall be applied perpendicular to the direction of moulding pressure.

Test shall be carried out in accordance with Annex E of IS 13411 : 1992 (Method A).

7.3 Determination of Temperature of Deflection Under Load

Two test specimens of length not less than 110 mm, of width 10 mm and of thickness 4 mm shall be used.

Test shall be carried out in accordance with Annex H of IS 13411 : 1992 (Method A).

7.4 Determination of Incandescence Resistance

Test shall be carried out in accordance with Annex C. After 3 minutes turn the ignition-bar holder away from the specimen and note whether there is any visible flame on the specimen during the next 30 seconds.

7.5 Determination of Insulation Resistance

Each test specimen shall be in the form of a flat plate moulded to a thickness of 3·0 mm ± 0·25 mm. The taper pin electrodes shall be used. Before carrying out the test, the specimen shall be conditioned (without electrodes) in an oven at 50°C ± 2°C for 24 h ± 1 h, and then cooled to room temperature in a desiccator. It shall then be immersed in distilled or de-ionized water at 27°C ± 2°C for 24 h ± 1 h. Before testing, the surface water shall be removed withb lotting or filter paper or with a clean absorbent cloth, and the electrodes fitted. The measurement of insulation resistance shall be made within 5 min after the end of the immersion. At least two specimens shall be used.

Tests shall be carried out in accordance with IS 2259 : 1963.

7.6 Determination of Electric Strength at Power Frequencies

At least two test specimens shall be used for the determination. Test specimen shall be 3·0 mm ± 0·25 mm thick and not less then 100 mm in diameter. Each test specimen shall be immersed in oil at a temperature of 90°C ± 2°C for 15 min to 20 min before the test and during the test. The oil shall preferably be one complying with the requirements of IS 335 : 1983. The 20 s step-by-step method shall be used.

Test shall be carried out in accordance with IS 2584 : 1963.

NOTE — It may be necessary to measure the short term value on an additional test specimen in order to determine the initial voltage to be applied.

7.7 Determination of Tracking Resistance Under Moist Conditions

Test shall be carried out in accordance with IS 2824 : 1975. The applied voltage shall be the numerical value of the comparative tracking index given in Table 1. Two determinations shall be made. The material shall not track.

7.8 Determination of Water-Absorption (Cold)

Two test specimens 50 mm ± 1 mm in diameter and 3 mm ± 0·2 mm thick shall preferably be used. As an alternative, when agreed between the purchaser and the supplier, square specimens of side 50 mm ± 1 mm cut from 4 mm ± 0·2 mm thick moulded plates may be used. If this alternative type of test specimen is used, the requirements shall also be the subject of agreement between the purchaser and the supplier.

Test shall be carried out in accordance with Annex D of 13411 : 1992 (Method 1).

7.9 Determination of Mould Shrinkage

Two test specimens shall be used. For material intended for injection or transfer moulding, the method of preparation of the test specimens shall be agreed to between the purchaser and the supplier.

Test shall be carried out in accordance with Annex B of IS 13411 : 1992.

7.10 Determination of Post Moulding Shrinkage

Two test specimens shall be used. For materials intended for injection or transfer moulding, the method of preparation of the test specimens shall be agreed to between the purchaser and the supplier.

Test shall be carried out in accordance with Annex B of IS 13411 : 1992.

8 FREQUENCY OF TEST

In determining compliance of a material with the requirements of this standard, it is usually appropriate to determine some properties more frequently than the others. For example, if the properties listed below for frequent determination are determined on one batch in ten batches, it may be sufficient to determine the remaining properties on one batch in fifty batches.

a) *Properties Requiring Frequent Determination*

   Bulk factor
   Volatile matter
   Flow
   Flexural stress at rupture
   Insulation resistance after 24 h in water
   Water absorption, cold
   Mould shrinkage

b) *Properties Requiring Less Frequent Determination*

   Impact strength, Izod

Temperature of deflection under load
Incandescence resistance
Electric strength
Comparative tracking resistance
Post shrinkage, 48 h

9 PACKING AND MARKING

9.1 Packing

The material shall be packed in well-closed containers or as agreed to between the purchaser and the supplier.

9.2 Marking

The containers shall be securely closed and legibly marked with the following information:

a) Indication of the source of manufacture, recognized trade-mark, if any;
b) Name and grade of the material;
c) Colour of the material;
d) Month and year of manufacture;
e) Batch No. or Code No.; and
f) Direction for storage and use.

9.3 BIS Certification Marking

The containers may also be marked with the Standard Mark.

9.3.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

10 SAMPLING

10.1 General

10.1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed:

a) Samples shall not be taken in exposed place.
b) The sampling instrument shall be made of stainless steel and shall be clean and dry.
c) Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.
d) Sample shall be placed in suitable, clean, dry, air-tight metal or polythene containers on which the material has no action.
e) Sample containers shall be of such a size that they are almost complety filled by the sample.
f) Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, such as the date of sampling, the month and year of manufacture of the material, the name of the material, etc.
g) Samples shall be stored in such a manner that the temperature of the material does not exceed 20°C.

10.2 Scale of Sampling

10.2.1 *Lot*

All the containers of urea formaldehyde moulding material of the same grade, manufactured under similar conditions of production shall be grouped together to constitute a lot.

10.2.1.1 *Number of samples*

For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out on each lot separately. The number of containers to be selected from the lot shall depend upon the size of the lot and shall be in accordance with Table 2.

Table 2 Scale of Sampling

Lot Size	No. of Containers to be Selected from the Lot
(1)	(2)
Up to 150	3
151 to 300	4
301 to 500	5
501 and above	7

Each container in the sample shall be selected at random from each lot. For this purpose, reference may be made to IS 4905 : 1968 'Methods of random sampling'.

10.3 Preparation of Test Samples and Referee Sample

10.3.1 *Sampling Instrument*

For sampling, instrument made of stainless steel shall be as used as shown in Fig. 2 (*see* page 9). It shall be capable of taking samples from all points when inserted in the container.

10.3.2 From each of the containers selected, representative portion of the material shall be drawn with the help of the sampling instrument. For this purpose, the material shall be taken from different points — atleast 75 mm away from any surface in the case of large containers, and 25 mm away in the case of small containers. The total quantity of the material collected from each container shall be sufficient for determinations of the various characteristics as specified in the relevant material specification (*see* **10.3.3** and **10.3.4**).

10.3.3 Out of these portions, a small but equal quantity of the material shall be taken out and mixed thoroughly to form a composite test sample. The composite test sample thus formed shall be divided into three parts and transferred to separate bottles. One of these bottles shall be marked for the purchaser, another for the supplier and the third for the referee.

10.3.4 The remaining portion of the material from each container shall be divided into three parts and each such part shall be transferred to separate bottles which are then sealed airtight with stoppers and labelled with full identification particulars of the sample. The material in each such sealed bottle shall constitute an individual test sample. These individual test samples shall be grouped into three sets in such a way that every set has a test sample representing each container selected. One of these three sets shall be marked for the purchaser, another for the supplier and the third for the referee.

10.3.5 *Referee Sample*

Referee sample shall consist of the composite test sample and a set of individual test samples marked for the purpose and shall bear the seals of the purchaser and the supplier and shall be used in the case of a dispute between the two.

10.4 Number of Tests

The test for description (**5.1**), Bulk factor [Table 1 (i)] and Flow [Table 1 (ii)] shall be made on each of the individual samples while test for all other characteristics shall be made on the composite sample.

10.5 Criteria for Conformity

10.5.1 *Individual Samples*

10.5.1.1 From test results for each characteristics tested on individual samples, the average ($\bar{X}$) and Range (R) (if sample size is less than 10) or Average Range ($\bar{R}$) (if sample is greater than or equal to 10), shall then be calculated.

$$\text{Average } (X) = \frac{\text{Sum of all test results}}{\text{Number of test results}}$$

Range (R) = Difference between maximum and minimum test results.

NOTE — For sample size 10 or more, range is calculated for five tests results taken in order in which they are obtained and then average range ($\bar{R}$) is computed.

The lot shall be considered as meeting the requirements of these characteristics, if:

> ($\bar{X} + 0{\cdot}6\,R$) or ($\bar{X} + 0{\cdot}6\,R$) is less than or equal to the maximum specified limit or ($\bar{X} - 0{\cdot}6\,R$) or ($\bar{X} - 0{\cdot}6\,\bar{R}$) is greater than or equal to the minimum specified limit for each of these characteristics.

10.5.2 *Composite Sample*

10.5.2.1 In respect of each characteristic tested on the composite sample, the lot shall be declared as conforming to the requirements of the specification if the test results on the composite sample satisfy the corresponding requirement.

10.5.2.2 The lot shall be declared as conforming to the requirements of the specification if **10.5.1** and **10.5.2** are satisfied.

ANNEX A

(*Clauses* 6 *and* C-4)

PREPARATION OF MOULDED TEST SPECIMENS

A-1 APPARATUS

A-1.1 Compression Mould

Made of steel, able to withstand the specified temperatures and pressures. The mould shall be designed so that the compression force is transmitted to the moulding material with no appreciable loss. It may be of a single-cavity or multi-cavity type. A semi-positive mould is also suitable, even though the pressure on the moulding material is not as well defined and in such cases, the specimen thickness shall be adjusted using spacers on the mould parting line.

The mould surface shall be free from superficial damage or contamination and have a shiny surface finish. Chrome plating is not always necessary, but it will prevent sticking.

The edge taper angle shall not be greater than 3°. Clearance between the vertical wall of the cavity and the punch shall not be greater than 0·1 mm.

The mould shall have a loading chamber large enough to allow the whole charge to be fed in one operation. Moulding materials in bulk form is from 2 to 10 times as voluminous as the moulded object.

The mould may be fitted with an ejector. If ejector pins are used, they shall not deform the specimens in any way. If the parts are ejected by the movable bottom of the mould, there shall be no significant leakage of material at the joint between bottom and the cavity wall.

Because the face of the moulded part facing the lower die is heated for a longer time during the period between filling and compression, it may be useful to distinguish between the two faces by means of a fixed mark in the cavity.

A-1.2 Heating Device

Capable of heating the mould so that the moulding temperature remains constant and uniform over all parts of the mould within the specified tolerances.

The mould may be heated either through the platen or by means of a built-in-device (for example, circulating fluid or electric heating elements). In the latter case, the mould shall be insulated from the press platens with a sheet of insulating material. For practical reasons, it is generally preferable to heat the mould electrically.

A-1.3 Mould Temperature Adjustment Device

Capable of ensuring that the optimum required temperature is maintained constant over the whole mould with a permissible deviation of ± 3°C, if the mould temperature shall not vary with time and position by more than ± 3°C.

A-1.4 Compression Moulding Press

Capable of ensuring that the specified pressure is applied and maintained during the whole of the curing time. The press may be hand operated or programmed.

It is preferable to use a press having two closing speeds:

- a fast approach speed (for example 200 mm/s to 400 mm/s) to avoid precure of the material before closing;
- a slow closing speed (for example 5 mm/s) to prevent air or gases from being entrapped.

NOTE — The oil pressure P_0 in MPa, to be applied, to obtain the specified pressure, P in MPa, is given by the equation:

$$P_0 = \frac{P \times A_1}{A}$$

where

A = area, in square metres of the press piston head; and

A_1 = total area, in square metres, of the cavities.

A-1.5 Stop Watch

Capable of being read to an accuracy of 1 s.

A-1.6 Mould Temperature Measurement Device

Such as a pyknometer or fusible salts.

A-1.7 Balance

Having an accuracy of 0 1 g.

A-1.8 Metal Plate

About 20 mm thick and having at least the same area as the specimen, for use as a cooling fixture after stripping.

A-2 PREFORMING

A-2.1 If the volume of the moulding material is too great for the capacity of the loading chamber of a conventional mould, the material may be preformed; the conditions used for such preforming shall be stated.

A-3 MOULDING CONDITIONS

A-3.1

Moulding Temperature, °C	*Moulding Pressure, MPa*	*Curing Time Per mm of Thickness, S*
140-150	15-35	30-60

If drying or preheating is carried out, the method, time and temperature shall be stated. In the case of preheating a curing time of 20-30 seconds per millimeter of thickness is permissible.

A-3.2 Drying

Materials may be dried prior to electrical tests. For drying, the material shall be spread out in a thin layer and heated for 60 min at 90°C±3°C. The material shall be moulded immediately upon removal from the oven.

A-3.3 High-Frequency Preheating

It is permissible as it permits a reduction in curing time. The preheated material shall be moulded immediately after preheating.

A-3.4 Pre-Plastification

It is permissible as it ensures thermal and mechanical homogenization of the material. The preplasticised material shall be moulded immediately after pre-plastification. For the conditions for the pre-plastification, an agreement shall be made between the interested parties and the conditions shall be stated.

A-3.5 Release Agents

Release agents that is products designed to facilitate the release of the moulding from the mould, may be used only if it has been proved that they have no influence on the moulded-specimen properties. This requirement applies only when the specimens are to be tested for electrical properties, spectroscopic analysis or adverse taste and colour.

A-3.6 Breathing

If it is necessary to open the mould for the purpose of breathing, it shall be reported.

A-4 PROCEDURE

Allow the moulding temperature to reach equilibrium at ± 3°C of the required value.

Check the temperature in the cavity using the temperature measurement device. Weigh out required quantity of material to obtain the specified thickness of specimen. This quantity is the product of the moulded-part density and the volume of the test specimens, to which is added flash losses as determined by previous testing. Load the material, as powder or preform, in the cavity. Close the press. Allow to breathe, if necessary. Start the stopwatch when the pressure has reached the specified value. When the curing time is completed, open the press. Remove the specimen immediately, and unless otherwise specified in the test method (*see* Note 2), allow it to cool on the metal plate used as the cooling fixture.

Check that the moulding is satisfactory as regards filling of the mould, appearance, absence of porosity, discoloration, flash and warpage. If necessary, check the density.

NOTES

1 In the case of a programme controlled press, degassifying and opening may be performed automatically.

2 For some test methods, such as the determination of shrinkage, the requirement may be to place the hot moulding on a material of low thermal conductivity and under an appropriate load.

ANNEX B

[*Table* 1, *Sl No.* (*A*, iii)]

DETERMINATION OF VOLATILE MATTER

B-1 APPARATUS

B-1.1 Wide Mouthed Weighing Bottles

Of internal diameter 45 mm and depth 35 mm, provided with tight-fitting covers.

B-1.2 Well-Ventilated Oven

Capable of being controlled at 105 ± 1°C.

B-1.3 Balance

With an accuracy of 0·001 g.

B-2 PROCEDURE

B-2.1 Weigh 5 ± 0·1 g of the sample to the nearest 0·001 g into a clean dried weighing bottle and spread evenly over the bottom of the weighing bottle. Conduct the test in duplicate.

Place the bottles, with covers removed, in the oven controlled at 105 ± 1°C. After a period of 1 hour, close the weighing bottle, remove from the oven and place in a desiccator at room temperature. After a period of at least 1 h, ease the covers of the weighing bottle to equalize pressure, and reweigh the bottles.

B-3 CALCULATION

The volatile matter in percent by mass, is given by the formula:

$$\frac{m_1}{m_0} \times 100$$

where

m_0 = original mass of the test portion; and

m_1 = loss in mass of the test portion.

Calculate the arithmetic mean of the two determinations.

ANNEX C

[*Table* 1, *Sl No.* (*B, iv*) *and Clause* 7.4]

DETERMINATION OF INCANDESCENT RESISTANCE

C-1 PRINCIPLE

Supporting of a test specimen horizontally by one end. Assessment of the flammability characteristics of the test specimen after bringing its free end into contact, for a specified period of time, with a silicon carbide rod electrically heated to a temperature of 955 ± 15°C.

C-2 SIGNIFICANCE OF THE TEST

> *Important* — This method must not be used to assess potential fire hazards in use.

Tests made on materials under the conditions described in this method can be of considerable value in comparing the flammability characteristics of various materials under the precise conditions of the test, in checking manufacturing processes, or as a measure of the deterioration or change in flammability prior to or during use. The test is designed for quality control and product evaluation and must not be regarded as significant for the behaviour of materials under conditions other than those specified in the test; in particular, it is not predictive of behaviour in real fire situations.

C-3 APPARATUS

C-3.1 Test Chamber

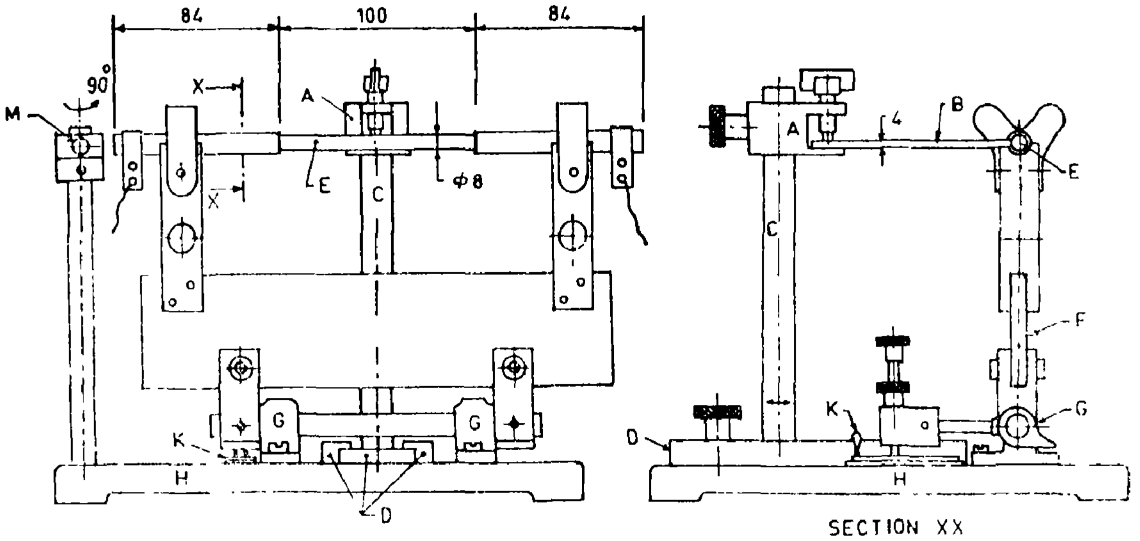
Draught-free, with a capacity of approximately 1 m^3, and permitting observations. For safety and convenience, it is desirable that this enclosure (which can be completely closed) be fitted with a device, such as an exhaust fan, to remove products of combustion which may be toxic. However, it is important to note that the device must be turned off during the actual test and started again immediately after the test to remove the products of combustion.

C-3.2 Test Unit

Comprising the following items (*see* Fig. 1).

C-3.2.1 *Clamp* (*A*)

To fix the test specimen (*B*), as shown in the Fig. 1 (section (*X X*), mounted on top of an upright holder (*C*), provided with a sliding base (*D*), so that specimens of different lengths can be tested.



All dimensions in millimetres.

FIG. 1 TEST UNIT

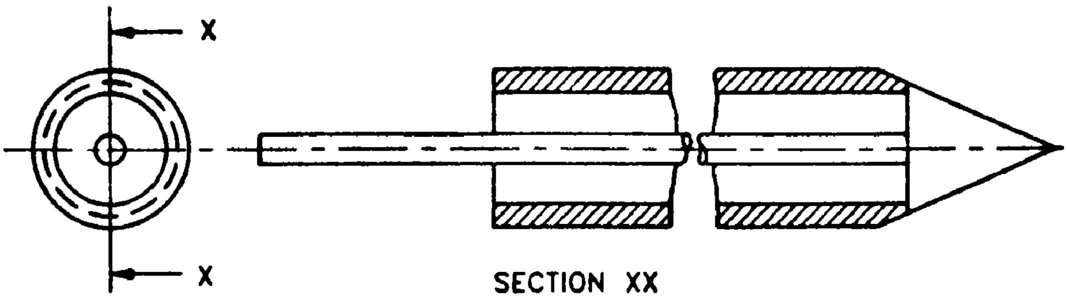


Fig 2 Sampling Instrument

C-3.2.2 *Igniting Source*

A bar consisting of a silicon carbide rod (*E*) of diameter 8 mm with a usable length of about 100 mm and metallized contact ends. The rod shall be capable of being heated to a temperature of 955 ± 15°C by alternating current or by direct current using, respectively, a regulating transformer or a rheostat. A suitable voltmeter and ammeter or wattmeter shall be used to adjust the electrical output to about 350 *W*.

C-3.2.3 *Insulated*

Suitable (ceramic or asbestos material) holder (*F*) in which the silicon carbide rod (*E*) is mounted, arranged to rotate about a horizontal axis in two bearings (*G*), fixed on the base plate (*H*). By this means the silicon carbide rod may be removed completely away from the test specimen (*B*), when necessary. The dimensions of the base plate (*H*) are approximately 275 mm × 250 mm.

C-3.2.4 *Counterweight* (*I*)

Dimensioned so that the incandescent rod (*E*) exerts a force of about 0·3 N on contacting the test specimen (*B*).

C-3.2.5 *Stop Screw* (*J*)

Placed by means of the pivoting plate (*K*) so as to permit the incandescent rod (*E*) to remain in contact with the test specimen (*B*) until about 5 mm of the latter has been burnt away.

C-3.2.6 *Pivoting Metal Rod* (*M*)

Of diameter 8 mm and length 150 mm, to locate before the test, the front end of the clamped specimen in a flatwise position as shown in the Fig 1 (Section *X X*). This rod shall be capable of being rotated into the exact position occupied by the incandescent rod during the test.

C-3.3 Means

Of checking the temperature of the incandescent rod.

C-3.4 Stop-Watch

C-4 TEST SPECIMENS

Five test specimens, each measuring 10 mm × 4 mm × 80-130 mm in length shall be tested. Each specimen shall be marked by a line perpendicular to the longitudinal axis of the bar, 95 mm from the end which is to be burnt. The line can be engraved into the surface of the test specimen. Test specimens shall be prepared in accordance with Annex A of the standard.

Unless otherwise specified or agreed upon by the interested parties, specimens shall be tested in the 'as received' condition.

C-5 PROCEDURE

C-5.1 Adjustment of the Test Unit

Tilt the holder (*F*) with the rod (*E*) down and away from its normal position to permit the metal rod (*M*) to be turned into the position to be occupied by the incandescent rod during the test. Set up the specimen so that the distance between the clamp (*A*) and the line on the test specimen (*see* **C-4**) is approximately 10 mm. Adjust the clamp (*A*) and the upright holder (*C*) to locate the front end of the specimen in contact with the metal rod (*M*).

C-5.2 Ignition

Heat the rod (*E*) to a temperature of 955 ± 15°C in its tilted-down position. When the temperature reaches the correct value and remains constant (that is, when the readings of the ammeter or the wattmeter become constant) turn the metal rod (*M*) back to its original position, and tilt the holder (*F*) up so that the incandescent rod comes into contact with the front end of the specimens. After 3 min, remove the incandescent rod from the specimen by rotating the support (*F*). Determine the burning time, *t*, in seconds, using the stop-watch, starting with the first appearance of flames and ending with the disappearance of flames or when they reach the 95 mm mark.

C-5.3 Termination of Test

C-5.3.1 If no flame is visible on the specimen, terminate the test 30 s after the incandescent rod has been removed.

C-5.3.2 If a specimen stops burning before the flame front reaches the line (95 mm from the end to be burnt), terminate the test, but not until 30 s after the incandescent rod has been removed.

C-5.3.3 If the flame reaches the line, terminate the test and extinguish the flame.

C-5.4 Additional Measurement

After the test is terminated, measure the smallest undestroyed distance between the line and the nearest destroyed area on all surfaces of the specimen. Record the smallest of these measurements of undestroyed length, p, in millimetres.

The undestroyed length shall be taken as the distance from the line to the nearest evidence of damage to the test specimen due to flame impingement, including areas of complete or partial consumption, charring or embrittlement, but not including areas sooted, stained, warped or discoloured, nor (where this can be assessed with confidence) where the material has shrunk or melted away from the heat source.

C-6 EXPRESSION OF RESULTS

Calculate

- the average burning time, t, in seconds, of the five test specimens;
- the average destroyed length, L, in millimetres, of the five test specimens.

For an individual specimen

$$L = 95 - p$$

C-7 TEST REPORT

C-7.1 The test report shall contain the following particulars:

i) Full identification of the product tests: type, source and manufacturer's number and code;

ii) Description of the preparation of the test specimen and, if applicable, conditioning of the test specimens;

iii) The average burning time, t, in seconds;

iv) The average destroyed length, L, in millimetres; and

v) Any special observations made during the test (how specimens ignited, smoke formation and color, whether there was melting without burning, glowing, falling burning particles, or sagging or buckling of the burning section, any unusual behaviour of the test specimens, etc).

C-7.2 The test report shall also include the following statement.

> *Important* — These test results relate only to the behaviour of test specimens under the particular conditions of the test; they are not intended, and must not be used, as a means of assessing the potential fire hazards of the material in use.

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